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TECHNICAL NOTE 3402

BOUNDARY LUBRICATION OF STEEL WITH FLUORINE- AND
CHLORINE-SUBSTITUTED METHANE AND ETHANE GASES

By S. F. Murray, Robert L. Johnson, and Max A. Swikert

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Cleveland, Ohio



Washington
February 1955

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SUMMARY

A study was made of the lubrication of steel by a series of stable fluorine- and chlorine-substituted methane and ethane derivatives. Several compounds containing fluorine and two or more chlorine atoms per molecule functioned as boundary lubricants to reduce friction and to prevent surface welding and metal transfer; stable fluorine compounds containing no chlorine did not prevent surface failure and therefore were not lubricants. Proper run-in was necessary to prevent initial surface failure with chlorine-substituted gases.

Difluorodichloromethane, tetrafluorodichloroethane, and other gases lubricated almost as well as conventional liquid lubricants. Difluorodichloromethane lubricated steel well enough at 480° F to prevent excessive wear, surface failure, and metal transfer.

The method and conditions of application are very critical in lubrication by gaseous materials. Further research data are necessary in order to specify means for assuring successful gaseous lubrication in practical mechanisms.

INTRODUCTION

Future demands in gas-turbine engines for aircraft will result in higher operating temperatures for bearings and lubricants than those encountered at present (refs. 1 to 4). To date, synthetic oils have shown the most promise as lubricants for the immediate future, but use of liquid lubricants at bearing temperatures much above present values will be limited by the oxidation of such fluids.

Several means of extending the temperature range of effective lubrication have been suggested, among them are the use of:

- (1) Stable fluids such as those polynuclear aromatic compounds that are derivatives of naphthalene (ref. 5)

- (2) Solid lubricants, molybdenum disulfide and graphite (ref. 6)
- (3) Externally pressurized air bearings (ref. 7)
- (4) Closed lubrication systems to reduce contact of fluid lubricants with air (ref. 8)

This paper describes an investigation of another possible solution to this problem: the use of halogenated gases as lubricants.

Tetrafluoromethane is a stable halocarbon as it does not show any measurable decomposition rate below 2000° F (ref. 9). Although partial substitution of chlorine for fluorine in this compound produces compounds having decomposition temperatures considerably lower, the fluorochloromethanes so formed are quite stable thermally and chemically. Experiments were made to find a compound of this type that would be chemically inert except at points of contact between sliding steel surfaces. At such points, flash temperatures induced by high stresses would be high enough to decompose any adsorbed molecules of gas and liberate enough chlorine to react with the steel surfaces. Such a reaction would form a low-shear-strength iron chloride film. The lubricating effectiveness of iron chloride films has been established by many investigators (refs. 10 and 11); the use of organic compounds containing chlorine and other active atoms is common lubrication practice for extreme loading conditions.

The investigation was carried out at the NACA Lewis laboratory. A kinetic-friction apparatus having a hemispherical mild-steel specimen sliding on a rotating mild-steel disk was operated in an atmosphere of the gas under investigation. After a brief run-in period at low speeds and light loads, friction force was measured at a sliding velocity of 120 feet per minute and with a load of 1200 grams (initial Hertz surface stress 158,000 psi). Most data were obtained at room temperature; however, some runs were made at bulk lubricant temperatures up to 480° F. Experiments were made with a series of the most stable halogenated methane and ethane derivatives. For purpose of comparison, a trifluorochloroethylene polymer and a mineral oil (liquids) were also investigated.

APPARATUS AND PROCEDURE

The apparatus used, described in detail in reference 12, is shown schematically in figure 1. The basic elements are a rotating disk specimen (SAE 1020 steel; hardness, Rockwell A-50; $2\frac{1}{2}$ in. diam.) and a hemisphere-tip rider specimen (also SAE 1020 steel; tip radius, $3/16$ in.). All specimens were made from the same lot of steel. During a run, the stationary rider slides in a continuous circumferential path on the flat

surface of the rotating disk specimen. The disk is rotated through a belt system by an electric motor coupled to a variable-speed power transmission unit. For the gaseous lubricants, a 2-liter Inconel pot, with several strip heaters mounted on the outside wall and a concentric set of ring heaters underneath, was used in place of the pyrex jar shown in figure 1. Loading was applied to the rider specimen by the use of dead weights acting through the pulley system. The loads used in this investigation were between 200 and 1200 grams (initial Hertz surface stresses, 87,000 to 158,000 psi). Friction force between the specimens was measured by means of four strain gages mounted on a copper-beryllium dynamometer ring; the strain-gage readings were registered on an indicating-type calibrated potentiometer. Repetitive tests indicated that friction coefficient values were generally reproducible to within ± 0.02 . Fluid temperatures were measured by means of a chromel-alumel thermocouple (contained in a stainless-steel sheath) mounted beside the rotating disk; the readings were recorded by a potentiometer. For each temperature condition, this thermocouple was calibrated against a thermocouple installed in the rider specimen. The temperatures reported herein are bulk specimen temperatures corrected for radiation effects by means of previous calibration.

The method of finishing specimens included rotation in a drill press while the surface was rubbed with successively finer grades of abrasive cloth. Grade 1/2 polishing cloth followed by a brief rubbing with crocus cloth produced the final finish of the disk specimens. This procedure left uniform circumferential finishing marks on the disk; surface roughness, as measured with a profilometer, was approximately 15 to 17 rms. The rider specimens were polished with grade 3/0 emery paper. Since preliminary experiments with various cleaning methods showed that any contaminating films left on the specimens after the polishing procedure had no discernible effect on friction measurements, no cleaning was given after the finishing, except for a brief washing with distilled acetone just prior to testing. Since the rider continuously slides over the same track on the rotating disk, the influence of minor contaminating films is minimized.

The experimental compounds and some of their properties are listed in table I. The gases were commercial-grade materials having purities higher than 97 percent. The impurities were primarily noncondensable gases, with less than 0.0025 percent water. In these experiments, gas was piped from the cylinder to the experimental apparatus through Tygon tubing and a stainless-steel elbow tube that extended from the top almost to the bottom of the Inconel pot. Several runs were made using copper instead of Tygon tubing, but no effect of materials was observed. Gas flow was measured by means of a calibrated flowmeter. The rate of flow was 4 liters per minute. An analysis of the atmosphere in the pot in runs using difluorodichloromethane and tetrafluorodichloroethane showed that the oxygen content was less than 0.5 percent after 5 minutes at

this flow rate (equivalent to changing the atmosphere of the pot ten times). Since this flow rate was maintained at all times during the test runs, contamination from the air was assumed to be negligible.

During tests of liquid lubricants, the specimens were submerged in the lubricant contained in a pyrex jar.

An unused set of specimens was used for each test. Gas flow was started 5 minutes before the test in order to displace the air. A standard run-in procedure, which is discussed further in the EXPERIMENTAL RESULTS section, followed purging of the container. A friction run comprised 60 minutes of continuous operation; readings were taken at random time intervals unless some change in conditions occurred. Runs were made at a sliding velocity of 120 feet per minute with a load of 1200 grams (initial Hertz surface stress, 158,000 psi). Most runs were made at room temperature; however, special runs were made with temperatures up to 480° F.

EXPERIMENTAL RESULTS

Run-in procedure. - Preliminary runs to determine suitable test conditions were made using difluorodichloromethane, a common refrigerant gas, as the lubricant. Operation at low loads and low rotative speeds gave promising results. At a high load, however, severe failure of previously unused specimens occurred between the surfaces during the first few revolutions. As running was continued at the higher load, friction force decreased gradually from the initially high value, and there was a transition from friction instability to smooth sliding. The severity of this initial run-in resulted in a wear scar of large diameter; the wear was caused by lack of lubrication until a reaction film was built up on the surface. This initial surface damage which obscured the lubricating effect of the gas could be prevented by increasing load gradually. Several runs were made to study this effect, and the following run-in procedure was evolved: At a constant speed of 55 feet per minute, the specimens were run with the following arbitrary loads and times: 200, 400, and 600 grams for 1 minute each, and 1200 grams for 2 minutes.

Presentation. - The results of the experiments are presented in figures 2 to 5 and also in table II, which presents a summary of the results obtained.

Friction measurements are presented as values of coefficient of friction μ , the ratio of restraining force to load. As bases for evaluating lubricating effectiveness of the various materials, the following observations were considered most important:

- (1) Value of coefficient of friction

- (2) Behavior of the specimens during the test. Instability of friction force and audible chatter of the specimens indicated ineffective lubrication
- (3) Condition of the surfaces after the test
- (a) No evidence of surface failure: effective boundary lubrication
 - (b) Evidence of some surface welding: incipient failure
 - (c) Evidence of extensive welding and metal transfer: mass failure
 - (d) Diameter of wear scar

The data presented are for runs that are representative of the results obtained in 2 to 12 runs on each variable.

Air. - Runs were made in air for comparison with the halogenated gases. The standard run-in procedure was followed, but because of high friction values (0.58-0.64) and audible chatter of the specimens, the test was stopped as soon as the speed was raised to 120 feet per minute after the run-in procedure had been completed. Wear and surface damage were severe. Figure 2(a) is a photomicrograph of the wear scar on the rider specimen, which showed evidence of severe welding.

Argon. - In order to check the effect of an inert atmosphere, argon gas was substituted for air; friction force (see table II) and wear-scar diameter were essentially the same as for air.

Tetrafluoromethane. - The fully fluorinated methane did not lubricate even during run-in. Severe welding and surface damage occurred and the coefficient of friction was between 0.57 and 0.67 (table II).

Trifluorochloromethane. - Trifluorochloromethane, although a poor lubricant, was slightly better than air (table II). Wear was high and some welding was apparent, but there was visual evidence of a film having been formed on the surface.

Difluorochloromethane. - Similar results were obtained with difluorochloromethane (table II), which contains the same number of chlorine atoms per molecule as trifluorochloromethane.

Difluorodichloromethane. - Difluorodichloromethane gave effective lubrication under the conditions of these tests with friction coefficients ranging from 0.125 to 0.170 (table II). Friction values observed are shown in figure 3(a) which shows the variation in coefficient of

friction during the 60-minute run. A photomicrograph of the rider wear scar is shown in figure 2(b). Chemical analyses of the films that covered the wear scar on the rider specimens showed qualitatively that a chloride film had been formed.

To determine whether difluorodichloromethane was an effective lubricant at higher temperature also, a series of 60-minute runs was made at a specimen temperature of $480^{\circ}\pm 20^{\circ}$ F. Difluorodichloromethane was selected for the high-temperature runs because it is one of the most stable compounds that was an effective lubricant in the room-temperature runs. The operating temperature of 480° F is in the range of interest for aircraft turbine-engine bearings. The specimens used in high-temperature runs were first run-in at room temperature, and were also run for 5 minutes at 120 feet per minute with a load of 1200 grams before the temperature was raised, to ensure the preliminary formation of an effective film on the surfaces. Friction values observed during the high-temperature runs are shown in figure 3(b). The coefficient of friction (0.32 ± 0.02) was higher than at room temperature but the surfaces appeared to be effectively lubricated; no surface failure or metal transfer was found, and the wear-scar diameter was only slightly larger than that produced at room temperature. Presence of a small amount of water vapor, deliberately introduced into the pot, lowered friction to the values obtained in the room-temperature runs. This phenomenon warrants further investigation.

Fluorotrichloromethane. - Since fluorotrichloromethane has a boiling point of 75° F, the gas inlet tubing and the specimens were kept at a constant temperature of about 100° F in order to prevent condensation. Although the wear-scar diameter and surface appearance of the friction specimens were comparable to the specimens run in difluorodichloromethane, the coefficient of friction was significantly higher (table II).

Carbon tetrachloride. - Runs were made with carbon tetrachloride both as a gas and as a liquid. For the gas runs, carbon tetrachloride was boiled from a flask into the pot through a heated glass inlet tube, and the specimens kept at about 200° F. Since it was desirable to purge most of the air out of the pot before beginning the run, the pot and the head of the flask were brought up to temperature. The carbon tetrachloride was then heated and allowed to boil for 8 minutes before the test was started. The results obtained in the tests are listed in table II. Although the appearance of the running surfaces and the wear-scar diameters were essentially the same for the gas as for the liquid, coefficient of friction was appreciably lower for the gas.

Halogenated ethanes. - Two ethane derivatives, tetrafluorodichloroethane and difluoroethane, were also tested. Tetrafluorodichloroethane gave very nearly the same results for friction and wear (table II) as difluorodichloromethane.

Difluoroethane failed during the lightest load of the run-in (table II). The contact surfaces showed severe welding and damage similar to that observed with tetrafluoromethane.

Liquids. - In order to evaluate the results obtained with the gases, two liquid lubricants were run at room temperature for comparison. The friction results for the first fluid, a trifluorochloroethylene polymer, are shown in figure 4(a). For this test, a 60-minute run was made with a load of 1200 grams at a speed of 120 feet per minute. The specimens were run-in by the standard procedure in order to make the results strictly comparable. A photomicrograph of the wear scar on the rider is shown in figure 5(a).

Runs were also made with grade 1010 mineral oil as the lubricant, in order to have a lubricant of known characteristics as a standard for comparison. Tests were run for 60-minutes after the standard run-in procedure. The friction results of a typical run are plotted in figure 4(b). A photomicrograph of the wear scar is shown in figure 5(b). The wear-scar diameter produced was significantly lower for both liquids than for the gaseous lubricants tested.

DISCUSSION

The lubrication results obtained showed that of the materials tested, those containing the larger amounts of chlorine were, in general, the best lubricants. This relation would be expected, because continual formation of a reaction film of iron chloride is conducive to effective lubrication. Chemical tests showed qualitatively that a chloride film had been formed with difluorodichloromethane, which lubricated well.

Tetrafluoromethane, the most stable compound of this series, was the poorest lubricant. Trifluorochloromethane and difluorochloromethane lubricated somewhat better, but permitted considerable wear and surface damage. Difluorodichloromethane lubricated satisfactorily under the conditions of these tests.

A similar dependence of lubricating effectiveness on chlorine content was apparent with the two ethane derivatives. Tetrafluorodichloroethane, which is equal to difluorodichloromethane in the number of chlorine atoms per molecule, was an equally effective lubricant.

The reason that the friction values obtained with fluorotrichloromethane are so much higher than those obtained with difluorodichloromethane is not known. Another deviation from the results that might be expected was observed with carbon tetrachloride. Friction coefficient was much lower with the gas than with the liquid, although surface appearance and wear were the same for both phases. Shaw (ref. 13) found that

the coefficient of friction between a cutting tool and aluminum stock was essentially the same when carbon tetrachloride was used as the cutting fluid in either the vapor or the liquid state.

Consideration of these gases as practical lubricants requires information on the limiting temperatures at which they can be handled in bulk. As with liquid lubricants, it is possible that a gas can lubricate a bearing surface that is at a temperature higher than that at which oxidative or thermal degradation of the lubricant begins to occur. Although gases would not be as effective as liquids for bearing coolants because of their lower specific heats, they are stable materials at higher temperatures than conventional liquid lubricants are.

Decomposition of difluorodichloromethane (as measured by a slow flow of gas through a heated glass tube packed with a steel wool) occurs only at temperatures above 800° F (ref. 14). Decomposition of tetrafluoromethane, the most stable of the compounds investigated, becomes significant only at temperatures above 2000° F (ref. 9). Data on decomposition of the other materials tested are not available.

Preliminary experience showed that the type of bearing-surface material has considerable effect on the ability of gases to provide effective lubrication. Further, moisture availability, condensation of gases, contaminants, and probably other factors affect the application of gaseous lubricants. Also, deliquescence of the iron chloride reaction product can cause corrosion of the bearing surfaces.

SUMMARY OF RESULTS

Boundary lubrication of steel surfaces was studied by use of a sliding-friction apparatus. The steel surfaces were lubricated with fluorine- and chlorine-substituted methane and ethane derivatives. The following observations were made:

1. Two stable compounds containing fluorine and no chlorine, tetrafluoromethane and difluoroethane, were not effective lubricants; high friction, surface failure, and considerable wear resulted from their use.

2. Fluorinated compounds that also contained chlorine gave at least partial surface protection. Molecules containing only one chlorine atom operated with high values of friction and wear. Molecules with two or more chlorine atoms lubricated with acceptable values of friction, and prevented excessive wear and surface damage. With all gaseous lubricants, it was necessary to run-in the surfaces at light loads to prevent initial high wear and surface damage. Four of the gases, difluorodichloromethane, fluorotrichloromethane, carbon tetrachloride, and tetrafluorodichloroethane, gave lubrication results almost as good as those obtained with liquid lubricants.

3. Difluorodichloromethane lubricated steel at 480° F well enough to prevent excessive wear and surface damage.

4. These preliminary data indicate the possibility of using some of these gases as lubricants in high-temperature systems. Laboratory experience has shown that the bearing material, lubricant, and moisture availability as well as run-in procedure are very important to the ability of gases to provide lubrication. Further research data are necessary in order to specify means for assuring successful gaseous lubrication in practical mechanisms.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, December 20, 1954

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TABLE I. - EXPERIMENTAL LUBRICANTS

Lubricant	Specific gravity of gas (air=1)	Boiling point, °F	Structure
Air	----	-----	---
Argon	1.38	^a -301	A
Tetrafluoromethane	3.04	^b -198.4	$\begin{array}{c} \text{F} \\ \\ \text{F}-\text{C}-\text{F} \\ \\ \text{F} \end{array}$
Trifluorochloromethane	3.60	^b -114.7	$\begin{array}{c} \text{F} \\ \\ \text{F}-\text{C}-\text{Cl} \\ \\ \text{F} \end{array}$
Difluorochloromethane	2.98	^b -41.44	$\begin{array}{c} \text{F} \\ \\ \text{F}-\text{C}-\text{Cl} \\ \\ \text{H} \end{array}$
Difluorodichloromethane	4.17	^b -21.64	$\begin{array}{c} \text{F} \\ \\ \text{F}-\text{C}-\text{Cl} \\ \\ \text{Cl} \end{array}$
Fluorotrichloromethane	4.74	^b +74.78	$\begin{array}{c} \text{Cl} \\ \\ \text{F}-\text{C}-\text{Cl} \\ \\ \text{Cl} \end{array}$
Tetrachloromethane (carbon tetrachloride)	5.31 (gas)	^a +170	$\begin{array}{c} \text{Cl} \\ \\ \text{Cl}-\text{C}-\text{Cl} \\ \\ \text{Cl} \end{array}$
Tetrafluorodichloroethane	6.0 ^a 5.90	^b +38.39	$\begin{array}{c} \text{F} \text{ F} \\ \quad \\ \text{F}-\text{C}-\text{C}-\text{F} \\ \quad \\ \text{Cl} \text{ Cl} \end{array}$
Difluoroethane	2.28	^a -12.5	$\begin{array}{c} \text{F} \text{ H} \\ \quad \\ \text{F}-\text{C}-\text{C}-\text{H} \\ \quad \\ \text{H} \text{ H} \end{array}$
Trifluorochloroethylene polymer	----	^c Viscosity, 4.13 cs at 100° F	$\left[\begin{array}{c} \text{F} \text{ F} \\ \quad \\ -\text{C}-\text{C}- \\ \quad \\ \text{F} \text{ Cl} \end{array} \right]_n \text{ (d)}$
Grade 1010 turbine oil	----	^c Viscosity, 9.95 cs at 100° F	Petroleum base stock

^aRef. 15.^bRef. 16.^cMeasured data.^dChain endings probably derived from solvent; rigorously fluorinated.

TABLE II. - SUMMARY OF RESULTS (BASED ON FRICTION, WEAR, AND SURFACE DAMAGE)

Number	Lubricant	Relative lubricating effectiveness	Representative coefficient of friction ^a (Speed, 120 ft/min; load, 1200 g)	Representative condition of surfaces	Remarks on lubrication
1	Air	Poor	0.58-0.64	Mass failure	Mass failure during run-in at all loads and speeds.
2	Argon	Poor	0.53-0.65	Mass failure	Similar to air.
3	Tetrafluoromethane	Poor	0.57-0.67	Mass failure	Similar to air.
4	Trifluorochloromethane	Poor to fair	0.4-0.6	Incipient to mass failure	Some beneficial effect on wear and surface condition.
5	Difluorochloromethane	Poor to fair	0.4-0.6	Incipient to mass failure	Some beneficial effect on wear and surface condition.
6	Difluorodichloromethane	Good	0.13-0.17 0.32 at 480° F	Effective boundary lubricant	Friction higher at high temperature, no change in wear or surface damage.
7	Fluorotrichloromethane	Good	0.22 at 100° F	Effective boundary lubricant	Friction higher than 6, similar to 6 in wear and surface damage.
8	Carbon tetrachloride (liquid)	Good	0.23-0.4	Effective boundary lubricant	Friction high, wear and surface appearance similar to 6.
9	Carbon tetrachloride (gas)	Good	0.1 at 200° F	Effective boundary lubricant	Friction lower for gas than for liquid state. Wear and surface appearance similar to 6.
10	Tetrafluorodichloromethane	Good	0.15	Effective boundary lubricant	Similar to 6.
11	Difluoroethane	Poor	0.54-0.64	Mass failure	Similar to air.
12	Trifluorochloroethylene polymer	Good	0.12	Effective boundary lubricant	Wear-scar diameter slightly less than 6.
13	Grade 1010 turbine oil	Excellent	0.11	Effective boundary lubricant	Wear-scar diameter smaller than for any other lubricant tested.

^aAt room temperature unless stated otherwise.

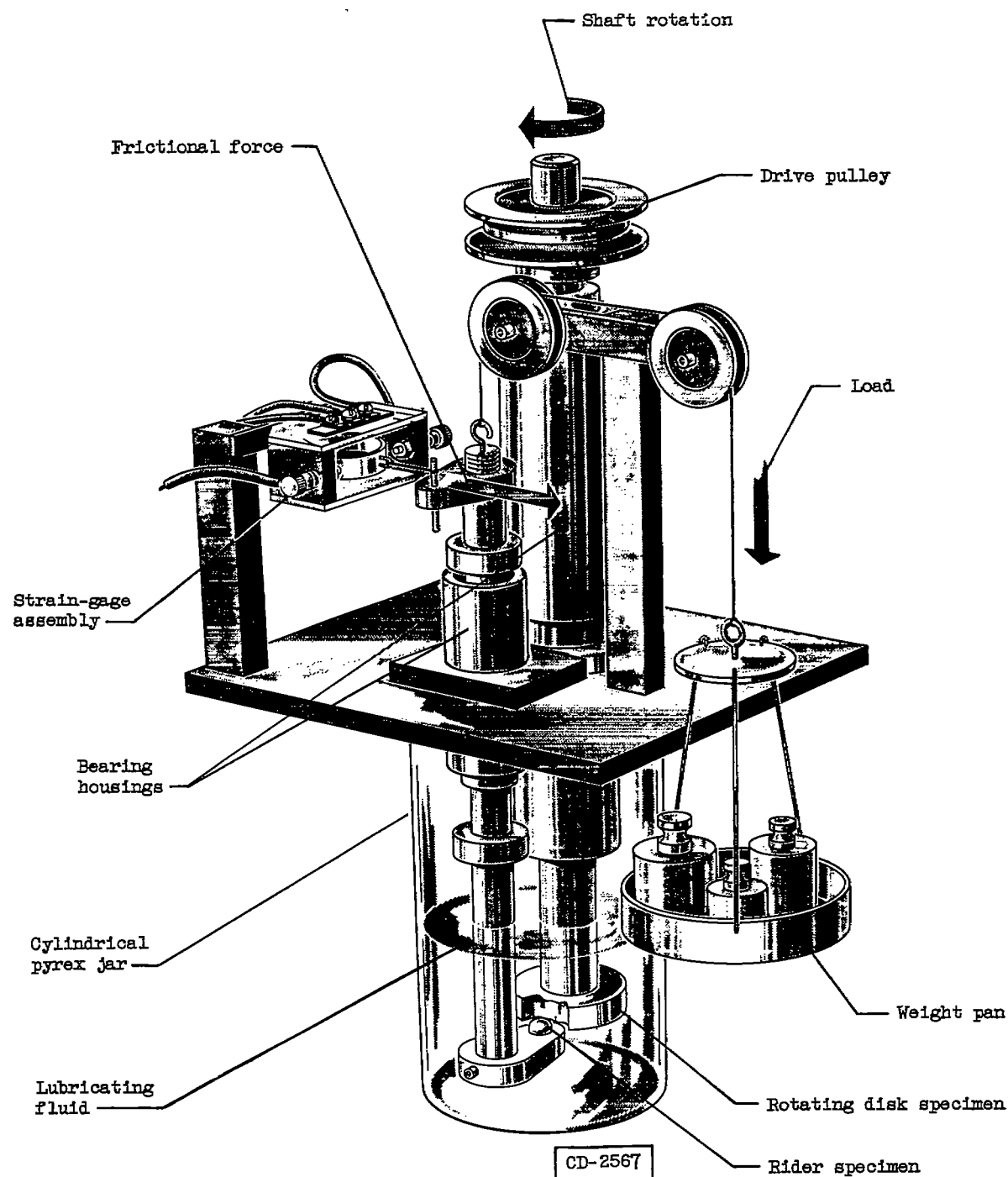
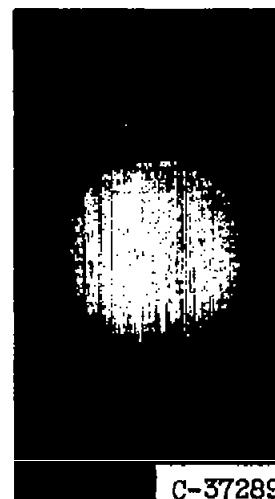


Figure 1. - Schematic diagram of friction apparatus for studying boundary lubrication by gases. The Pyrex jar shown was used for liquid lubricants; for gaseous lubricants, the jar was replaced with an Inconel pot.



(a) Mass failure. Steel against steel in air. After run-in attempt. X30.



(b) Effective lubrication. Steel against steel in difluorodichloromethane at room temperature. Time, 60 minutes; load 1200 grams; speed, 120 feet per minute. X30.

Figure 2. - Photomicrographs of wear scars on steel rider specimens that resulted from runs in air and in difluorodichloromethane.

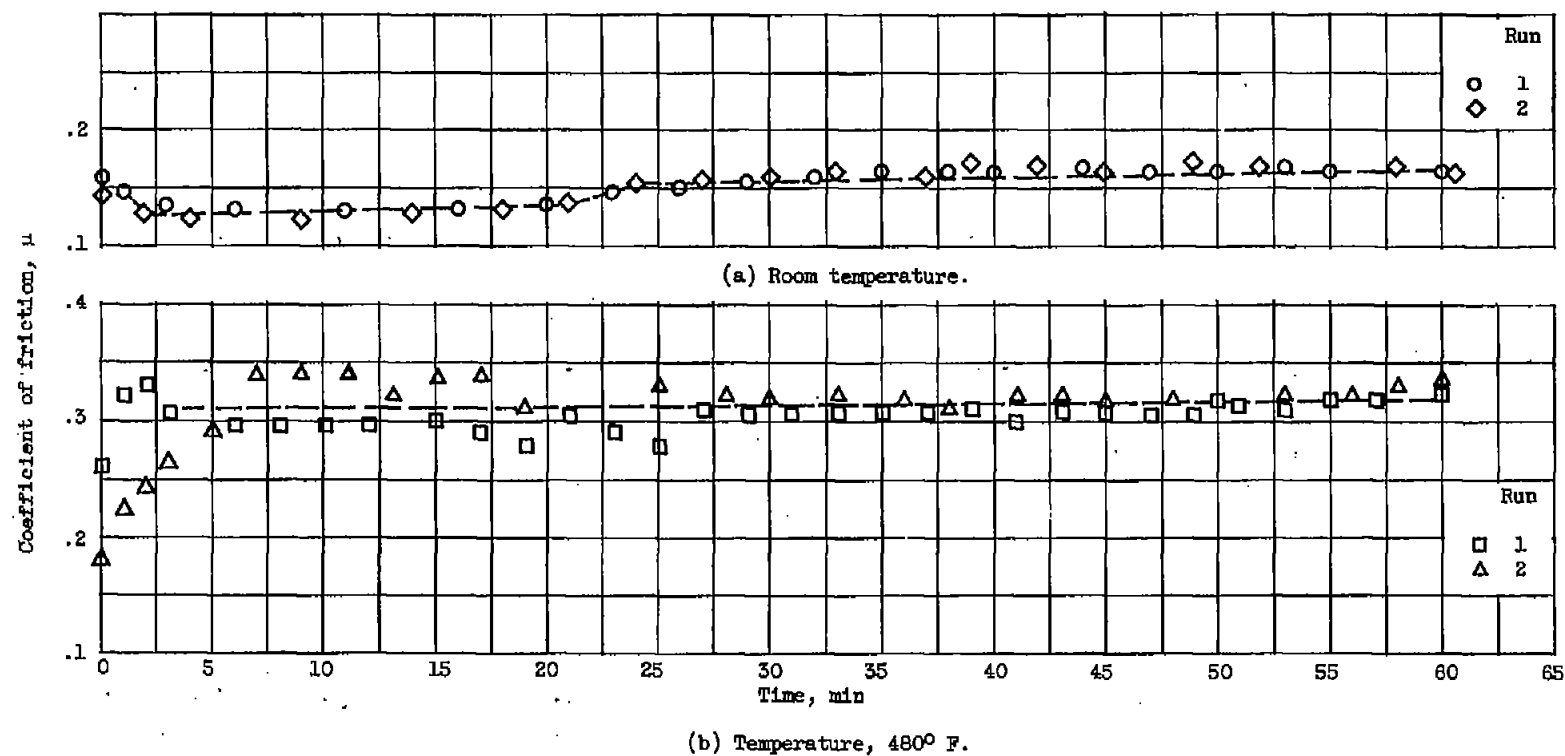


Figure 3. - Effect of time on the coefficient of friction of steel sliding against steel for two typical runs at different temperatures. Load, 1200 grams; speed, 120 feet per minute; lubricant, difluorodichloromethane.

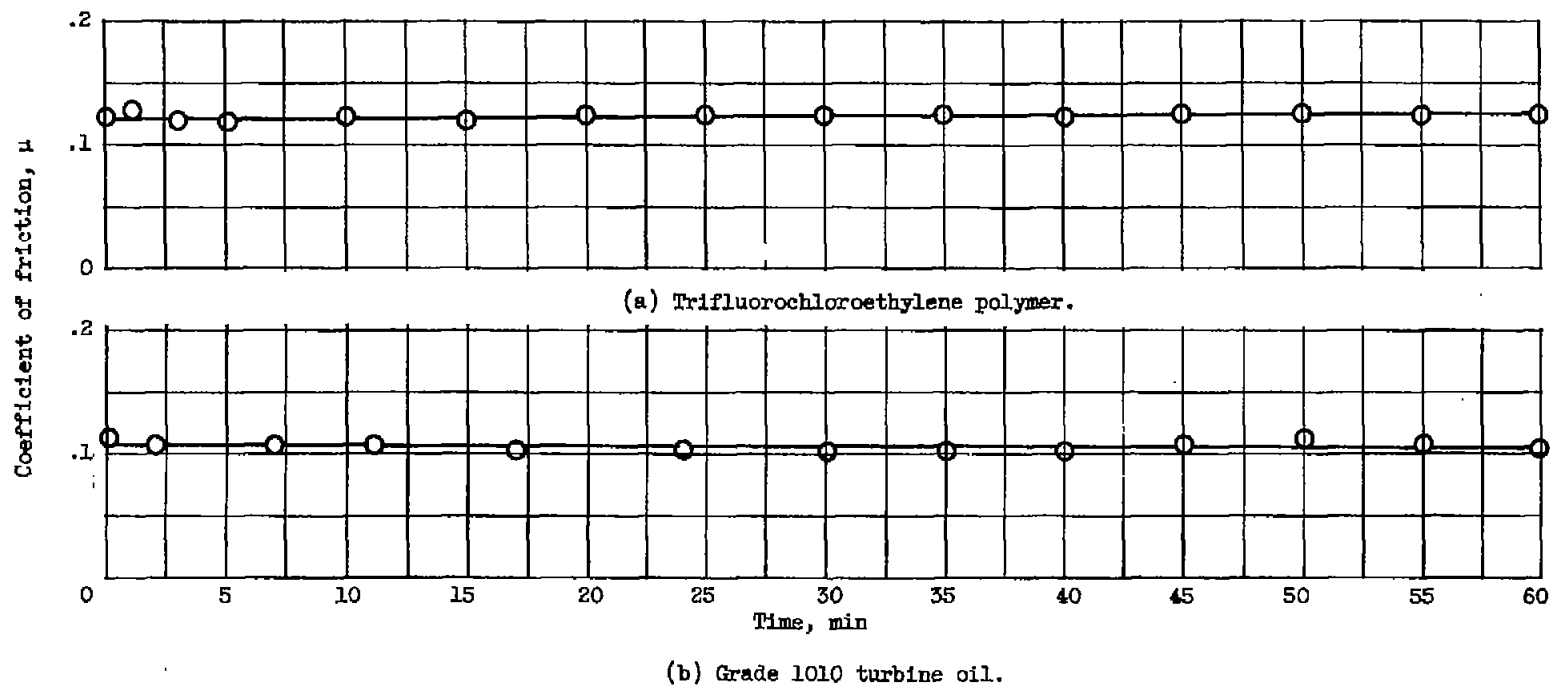
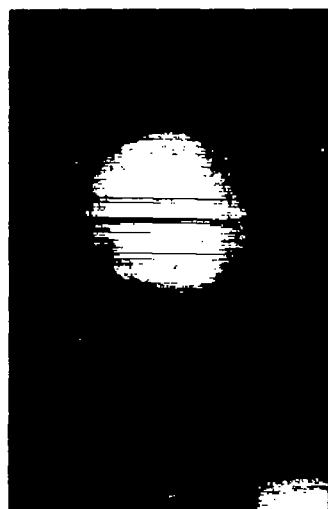
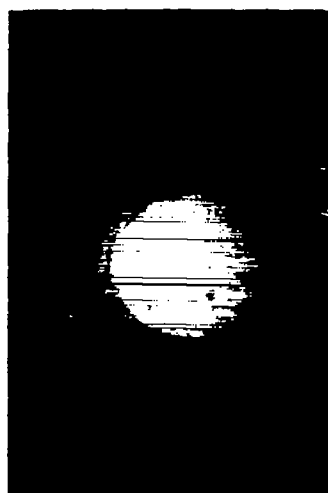


Figure 4. - Effect of time on the coefficient of friction of steel sliding on steel lubricated with liquid lubricants at room temperature. Load, 1200 grams; speed, 120 feet per minute.

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(a) Lubricant, liquid trifluorochloroethylene polymer.



C-37290

(b) Lubricant, grade 1010 turbine oil.

Figure 5. - Photomicrographs of wear scars on steel rider specimens obtained by running steel against steel with two different effective lubricants 60 minutes at a load of 1200 grams and a speed of 120 feet per minute. X30.